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Indian Standard

SPECIFICATION FOR DIBENZOTHIAZYL DISULPHIDE

(First Revision)

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MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

Gr 3 *July* 1989

Indian Standard
SPECIFICATION FOR DIBENZOTHIAZYL DISULPHIDE
(First Revision)

0. FOREWORD

0.1 This Indian Standard (First Revision) was adopted by the Bureau of Indian Standards on 5 January 1989, after the draft finalized by the Rubber Products Sectional Committee had been approved by the Petroleum, Coal and Related Products Division Council.

0.2 Dibenzothiazyl disulphide (MBTS) is a medium speed accelerator for vulcanization of rubber and finds a very wide range of applications in the rubber industry.

0.3 Dibenzothiazyl disulphide is insoluble in water, alcohol and petrol, slightly soluble in benzene and chloroform. It disperses readily in rubber under normal mixing conditions. Its solubility in rubber is approximately 0·25 percent (*m/m*).

0.4 This standard was first published in 1976. In this revision, requirements for coated MBTS, coating agent and manganese have been included while the requirement for relative density has been deleted. Further, of the two methods for determination of dibenzothiazyl disulphide, the simple chemical analysis method (Method A) has been designated as the referee method. Ultraviolet spectroscopy method has been deleted as it is very difficult to prepare pure chemicals for this analysis.

0.4.1 This standard also includes a recommended procedure for evoluating the material by means of a standard compounding and vulcanizing procedure.

0.5 This standard contains clauses **2.3** and **3.1** which call for agreement between the purchaser and the supplier.

0.6 For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS : 2-1960*. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

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*Rules for rounding off numerical values (*revised*).

1. SCOPE

1.1 This standard prescribes the requirements and methods of sampling and test for surface coated and uncoated dibenzothiazyl disulphide in powder form intended for use as an accelerator in rubber compounds.

2. REQUIREMENTS

2.1 Appearance – The material shall be in the form of a cream to light yellow fine powder having relative density of about 1·5 (for guidance only), and free from visible impurities.

2.2 The material shall also comply with the requirements given in Table 1 when tested according to the methods prescribed in col 4 of the table.

2.2.1 All the tests shall be carried out within one month of the date of receipt of the material by the purchaser.

2.3 Performance Requirement — The material when compounded and tested as given in Appendix B shall have its performance characteristics comparable to the sample complying with the requirements of **2.1** and **2.2** and previously approved by the purchaser.

3. PACKING AND MARKING

3.1 The material shall be packed as agreed to between the purchaser and the supplier.

3.2 The packages shall be securely closed and legibly marked to furnish the following information:

a) Manufacturer's name and registered trade-mark, if any;
b) Name of the material;
c) Net mass of the material in the package; and
d) Lot and batch number.

TABLE 1 REQUIREMENTS OF DIBENZOTHIAZYL DISULPHIDE

(*Clauses* 2.2 *and* 4.2.1)

Sl No.	Characteristic	Requirement	Method of Test, Ref to Cl No.
(1)	(2)	(3)	(4)
i)	Assay (MBTS content), percent by mass, *Min*		A-2
	a) Uncoated grade	97·0	
	b) Coated grade	95·5	
ii)	Free mercaptobenzothiazole (MBT content), percent by mass, *Max*	1·5	A-3
iii)	Surface coating (for coated grade only) percent by mass, *Max*	2·5	A-4
iv)	Melting point*:		A-3 of IS : 6918-1972†
	a) Appearance of first droplet	Not below 165°C	
	b) Completion of melting	Not below 171°C	
v)	Sulphated ash, percent by mass, *Max*	0·6	A-6 of IS : 6918-1972†
vi)	Residue on 150-micron IS Sieve, percent by mass, *Max*	0·75	3 of IS : 7086 (Part 1)-1973‡
vii)	Loss on heating for 2 h at 105°C, percent by mass, *Max*	0·6	7 of IS : 7086 (Part 1)-1973‡
viii)	Copper, ppm, *Max*	20	12 of IS : 7086 (Part 1)-1973‡
ix)	Manganese, ppm, *Max*	100	A-8 of IS : 6918-1972†

*In case of dispute in the melting point requirement, the final criteria for acceptance shall be the assay requirement.

†Specification for mercaptobenzothiazole.

‡Methods of sampling and test for rubber compounding ingredients, Part 1.

3.2.1 The packages may also be marked with the Standard Mark.

NOTE — The use of the Standard Mark is governed by the provisions of the Bureau of Indian Standards Act 1986 and the Rules and Regulations made thereunder. The Standard Mark on products covered by an Indian Standard conveys the assurance that they have been produced to comply with the requirements of that standard under a well-defined system of inspection, testing and quality control which is devised and supervised by BIS and operated by the producer. Standard marked products are also continuously checked by BIS for conformity to that standard as a further safeguard. Details of conditions under which a licence for the use of the Standard Mark may be granted to manufacturers or producers may be obtained from the Bureau of Indian Standards.

4. SAMPLING

4.1 For the purpose of ascertaining the conformity of dibenzothiazyl disulphide in a consignment to this specification, sampling as prescribed in **15** of IS : 7086 (Part 1)-1973* shall be followed.

4.2 Number of Tests

4.2.1 Test for copper (*see* Table 1) shall be conducted on individual samples.

4.2.2 Tests for all other characteristics shall be conducted on a composite sample.

4.3 Criteria of Conformity

4.3.1 *Copper* — The mean and range of test results for copper shall be calculated as follows:

$$\text{Mean } (\bar{X}) = \frac{\text{Sum of test results}}{\text{Number of test results}}$$

Range ($\bar{R}$) = Difference between the maximum and minimum value of the test results

The lot shall be deemed to have satisfied the requirement of the specified if

$$\bar{X} + 0{\cdot}6\,R \leqslant 20$$

4.3.2 *Composite Sample* — In respect of all other characteristics, the lot shall be considered as conforming to the specification if the composite sample satisfies each one of the requirements.

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*Methods of sampling and test for rubber compounding ingredients, Part 1.

APPENDIX A

[*Clause* 2.2 and *Table* 1, Sl No. (i), (ii) *and* (iii)]

TEST METHODS FOR DIBENZOTHIAZYL DISULPHIDE

A-1. QUALITY OF REAGENTS

A-1.1 Unless specified otherwise, 'pure chemicals' and distilled water (*see* IS : 1070-1977*) shall be employed in the tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

A-2. DETERMINATION OF MBTS CONTENT

A-2.0 General — Two alternative methods, namely, Method A and Method B for determination of MBTS content are given. However, in case of dispute, chemical analysis (Method A) shall be used.

A-2.1 Method A

A-2.1.1 *Principle* — In this method, MBTS is converted to MBT by reducing with hydrogen sulphide under alkaline conditions and following the iodomeric method for determining the resulting MBT.

A-2.1.2 *Reagents*

A-2.1.2.1 *Ethyl alcohol*

A-2.1.2.2 *Aqueous ammonia* — 25 percent

A-2.1.2.3 *Concentrated hydrochloric acid*

A-2.1.2.4 *Hydrogen sulphide gas*

A-2.1.2.5 *Iodine solution* — 0·1 N.

A-2.1.2.6 *Standard sodium thiosulphate solution* — 0·1 N.

A-2.1.2.7 *Starch indicator solution* — Triturate 5 g of starch and 0·01 g of mercuric oxide with 30 ml of water in a mortar. Pour the resulting paste into one litre of water and boil for three minutes, allow the solution to cool and decant off the clear liquid.

A-2.1.3 *Procedure* — Weigh accurately 0·4 g of the sample into a one litre conical flask with stopper. Add 80 ml of ethyl alcohol and 0·5 ml of ammonium hydroxide and reduce with hydrogen sulphide for 15 minutes, with occasional shaking. Expel the excess hydrogen sulphide by heating on a water bath and after acidifying with 1 ml of cocentrated hydrochloric acid, continue boiling for 20 minutes. Rinse the walls with 25 ml of ethyl alcohol and cool to room temperature. While shaking, add 25 ml of 0·1 N iodine solution and 500 ml of distilled water. Keep closed for 20 minutes and then titrate the excess iodine with 0·1 N sodium thiosulphate solution using starch as indicator. Carry out a blank under similar conditions.

A-2.1.4 *Calculation*

$$\text{MBTS, percent by mass} = \frac{(C-B)\times 1\cdot 66}{M} - \text{free MBT percent}$$

where

B = ml of standard sodium thiosulphate solution (0·1 N) required for sample,

C = ml of standard sodium thiosulphate solution (0·1 N) required for blank, and

M = mass in g of the sample taken.

A-2.2 Method B

A-2.2.1 *Principle* — In this method, MBTS is converted to MBT by reducing with hydrogen sulphide and following the iodometric method for determining the resulting MBT.

A-2.2.2 *Reagents*

A-2.2.2.1 *Ethyl alcohol*

A-2.2.2.2 *Hydrogen sulphide gas*

A-2.2.2.3 *Iodine solution* — 0·1 N.

A-2.2.2.4 *Standard sodium thiosulphate solution* — 0·1 N.

A-2.2.2.5 *Starch indicator solution* — as in **A-2.1.2.7**.

A-2.2.3 *Procedure* — Weigh accurately 0·4 g of the samples into a one litre conical flask with stopper. Add 80 ml of ethyl alcohol to dissolve the sample and reduce with hydrogen sulphide for 15 minutes with occasional shaking. Expel the excess hydrogen sulphide by heating on a water bath. Rinse the walls with 25 ml of ethyl alcohol and cool to room temperature. While shaking, add 25 ml of 0·1 N iodine solution and 500 ml of distilled water. Keep closed for 30 minutes and then titrate the excess iodine with 0·1 N sodium thiosulphate solution using starch as indicator. Carry out a blank under similar conditions.

A-2.2.4 *Calculation* — as in **A-2.1.4**.

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*Specification for water for general laboratory use (*second revision*).

A-3. FREE MERCAPTOBENZOTHIAZOLE (MBT) CONTENT

A-3.1 Reagents

A-3.1.1 *Iodine Solution* — 0·02 N.

A-3.1.2 *Standard Sodium Thiosulphate Solution* – 0·02 N.

A-3.1.3 *Starch Indicator Solution* — as in **A-2.1.2.7**.

A-3.1.4 *Chloroform*

A-3.2 Procedure

A-3.2.1 Weigh to nearest 0·1 mg, about 2 g of prepared sample into a stopper conical flask (500 ml) and dissolve in 100 ml of chloroform by slight warming. Cool and add 100 ml of water and 25 ml of 0·02 N iodine solution. Shake thoroughly and allow to stand for 3 minutes. Titrate the excess iodine with 0·02 N sodium thiosulphate solution adding approximately 2 ml of freshly prepared starch solution towards the end as indicator. Ensure that all the iodine is shaken out of the chloroform layer.

A-3.2.2 Carry out a blank test omitting only the sample.

A-3.3 Calculation

Mercaptobenzothiazole, percent by mass

$$= \frac{(B - A) \times 0·334}{M}$$

where

B = ml of standard sodium thiosulphate (0·02 N) solution used for the blank titration,

A = ml of standard sodium thiosulphate (0·02 N) used by the sample, and

M = mass in g of the sample taken.

A-4. DETERMINATION OF SURFACE COATING AGENT

A-4.1 Outline of the Method — In this method, the accelerator sample is washed with petroleum ether and filtered. The filterate of petroleum ether is evaporated to dryness over water bath. The content of petroleum ether soluble matter is taken as the content of surface coating agent.

A-4.2 Reagent

A-4.2.1 *Petroleum Ether* — boiling range 40 to 60°C.

A-4.3 Procedure — About 5 g sample of the accelerator is accurately weighed (M) to nearest 0·1 mg in an 100 ml Erlenmeyer flask. 25 ml of petroleum ether is added to the flask by means of a measuring cylinder. The contents of the flask are stirred thoroughly for five minutes. The contents are then filtered through a Gooch crucible (Type G4). The contents of the flask and the residue in the crucible are washed twice each time taking 25 ml of petroleum ether.

The filtrate and the washings are transferred to a previously weighed (W_1) accurate to nearest 0·1 mg evaporating dish. The petroleum ether is evaporated off over a water bath till a constant weight (W_2) is reached.

A-4.4 Calculation

Surface coating agent, percent by mass

$$= \frac{W_2 - W_1}{M} \times 100$$

where

W_1 = mass in g of the evaporating dish,

W_2 = mass in g of the evaporated dish and residue, and

M = mass in g of the sample taken.

APPENDIX B

(*Clause* 2.3)

METHOD FOR COMPOUNDING AND TESTING OF DIBENZOTHIAZYL DISULPHIDE

B-1. TEST COMPOUND

B-1.1 As a guidance, the following test compound may be used for testing performance properties of dibenzothiazyl disulphide in rubber compounds:

	Parts by Mass
Natural rubber, Grade ISNR5 (*see* IS : 4588-1986*)	100
Zinc oxide (*see* IS : 3399-1973†)	5
Stearic acid (*see* 1675-1971‡)	1
Barytes (*see* IS : 1683-1973§)	75
Sulphur	2·5
Dibenzothiazyl disulphide sample	0·5

B-2. PROCEDURE

B-2.1 Follow the procedure prescribed in NR: 9 of IS : 3660 (Part 1)-1972||.

B-3. TESTS

B-3.1 The tests given below are recommended for each test sample. The approved sample shall also be tested side by side using the same master-batch, that is, compound excluding sulphur and accelerator only.

B-3.1.1 Mooney scorch at 120°C in accordance with the method prescribed in NR : 8 of IS : 3660 (Part 7)-1988*.

B-3.1.2 Tensile strength, modulus at 300 percent elongation, elongation at break at difference cures 20, 30 and 40 minutes at 141°C (from below to above the expected optimum cure), in accordance with the method prescribed in IS : 3400 (Part 1)-1987†.

B-3.1.3 Hardness on optimum cure at 141°C, in accordance with the method prescribed in IS : 3400 (Part 2)-1980‡.

B-4. RESULTS

B-4.1 The values obtained with the test sample should not vary by more than ± 20 percent for Mooney scorch and ± 10 percent for all the other characteristics from those obtained with the approved sample.

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*Specification for rubber, raw, natural (*third revision*).

†Specification for zinc oxide for rubber industry (*first revision*).

‡Specification for stearic acid, technical (*first revision*).

§Specification for barytes for rubber industry (*first revision*).

||Methods of test for natural rubber, Part 1 (*first revision*).

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*Method of test for natural rubber: Part 7 Determination of Mooney viscosity (*second revision*).

†Methods of test for vulcanized rubber: Part 1 Tensile stress-strain properties (*second revision*).

‡Methods of test for vulcanized rubber: Part 2 Hardness (*first revision*).

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